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TESTS ON "COLD FUSION" WITH PALLADIUM/SILVER CATHODES

Report No. 91-59-K

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ABSTRACT

Results of tests on "cold fusion" in an electrochemical cell are reported. The tests were carried out on 75% Pd/25% Ag disc cathodes with 0.1M and 1M LiOD in D_2O electrolytes. No tritium was found in these tests and while there was some evidence of neutrons, the results are inconclusive because of possible electrical line interference problems.

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EXECUTIVE SUMMARY

TESTS ON "COLD FUSION" WITH PALLADIUM/SILVER CATHODES

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Considerable controversy has followed the claim made by Fleischmann and Pons of fusion at low temperature in an electrochemical cell consisting of a palladium cathode and an electrolyte of lithium deuteroxide in heavy water. The potential of this technique for electricity generation resulted in the initiation of preliminary laboratory tests which were reported previously.

The major findings reported in the literature are (i) the inability to reproduce the results by highly respected institutions with qualified personnel, sophisticated equipment and comprehensive test programs, (ii) the production of large quantities of tritium by a few laboratories but in only a fraction of the cells tested in each laboratory; reports by others of tritium being produced are declining (iii) variable production of neutrons from zero to a few standard deviations above background and (iv) the production of zero to 20% excess heat.

Palladium/silver alloy electrodes (discs ~ 1cm dia x 1.5mm thick) were investigated in this work. The cathode was heat treated under vacuum to remove residual hydrogen. Reliable tritium concentration data were obtained with the recognition of the possibility of chemiluminescence in tritium measurements.

No tritium activity was found in these tests even with the addition of poisons to inhibit recombination of D atoms to D_2 at the cathode surface. Similarly, cooling to near freezing temperature did not produce excess tritium or neutrons.

The neutron count rate exceeded background on six occasions but it was later determined that the signals could have been due to electrical line interferences. However, the frequency and low levels of neutrons generation, if any, suggest a low probability of usefulness of the technique in practice.

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Nuclear Systems and Integration

TESTS ON "COLD FUSION" WITH PALLADIUM/SILVER CATHODES

1.0 INTRODUCTION

The achievement of fusion at low temperature in an electrochemical cell has recently been claimed by Fleischmann and Pons(1989). The potential of this technique for electricity generation resulted in the initiation of laboratory tests with pure palladium cathodes which did not show any excess heat, tritium or neutron generation as reported previously/Cheh et al(1990)/. To investigate the effect further, it was decided to repeat the tests with cathodes of palladium/silver alloy; this material is used to make commercial permeation membranes for separation of hydrogen from impurities.

2.0 LITERATURE INFORMATION

To date, there have been many reports of heat, tritium and especially neutrons. The significant findings reported in the literature are:

HEAT

(a) Fleischmann and Pons' data suggest an excess heat of 50% based on the total energy input. It is estimated that 76 watts electricity and 74 watts thermal energy are produced for every 100 watts of electrical energy input to the cell. This assumes that 50% of the input power is electrical energy for electrolysis with the remaining 50% as ohmic loss and also that a fuel cell is used to produce electricity by recombining the D_2 and O_2 produced at 80% efficiency.

(b) Liaw et al(1990) used a molten salt fusion cell having Pd as the anode and aluminum as the cathode. The cell operated at about 370°C and therefore it would be useful for power generation. The LiD ionizes in the molten salts and the positive Li ion goes to the Al cathode and the negatively charged D goes to the Pd anode. High levels of excess power output were observed but the effect was sporadic. A significant amount ($\sim$ 14 times the background standard deviation) of excess helium-4 was detected in one of four samples of their Pd cathode.

NEUTRONS/PARTICLES

(c) Arata and Zhang(1990) reported intense neutron generation at a rate in excess of 10^8 n/s on electrolysing a large 2 cm diameter, 5cm long palladium cathode with LiOD in D_2O. Simultaneous signals were obtained in BF_3 and ^3He detectors. They measured large(30°C) spontaneous temperature excursions in the Pd cathode itself. Rapid deuterium desorption occurred when the temperature reached 110°C, the desorption resulting in a lowering of the Pd temperature and because of the hysteresis in the D/Pd ratio vs temperature curve, the temperature dropped to 80°C before the D was readsorbed. The bursts of heat are calculated to be about 20 watts; this phenomenon can explain the bursts of heat reported by Fleischmann and Pons but we do not think this is excess heat.

(d) The production of neutrons on fracturing of lithium deuteride crystals has been reported by Klyuev et al(1986) and may be a possible mechanism for the cold fusion observations.

(e) Yamaguchi(1990) produced large bursts of neutrons about three hours after placing in a vacuum a deuterium gas-loaded Pd wafer which was coated with gold on one side and manganese and oxygen on the other. The sample became deformed and the temperature apparently rose to 700°C. However this occurred in only one of twenty samples tested. Neutron bursts were not observed with hydrogen loaded controls. There are no reports of the work having been reproduced by others.

(f) Los Alamos/Claytor(1990)/ has achieved a milestone in their work on the gas/solid fusion systems. In both the Pd-Si-D and the Ti-D systems, they can reproducibly produce an effect. In the Ti-D system, clean Ti, deuterium and containers are required to reliably obtain neutrons in excess of background and this has been achieved in almost every cell. In the Pd-Si-D system, excess tritium is seen when integrated over several hundred hours of operation.

(g) Dickinson et al (1990) measured significant fast emission of negatively and positively charged particles, visible photons and long wavelength radio frequency signals accompanying the fracture of deuterated Ti. Slow emissions were also measured as a result of chemisorptive emission of charged particles as well as photons when the freshly exposed Ti surface reacted with active background oxygen. The intensity of the emissions was dramatically reduced with TiH.

TRITIUM

(h) Several laboratories including Bockris at Texas A&M and Storms at Los Alamos have reported the production of tritium. It appeared in less than 25% of the cells tested by each laboratory during early tests but there have been no reports recently. Dendrites were reported present in cells producing tritium. The high electric fields at the tips of dendrites may be responsible for high acceleration of D nuclei causing fusion but it is thought that distances are inadequate for high velocities to be obtained. The number of reports of tritium being produced are decreasing with time.

3.0 EXPERIMENTAL DETAILS

The main experimental equipment consisted of two identical electrochemical cells (Figure 1).

3.1 Electrodes

Both cathodes were made from an alloy of 77.5% Pd and 22.5% Ag. The cathode in each of the electrolysis cells was prepared by cutting bar stock to obtain a 1.35 cm dia x 1.6mm thick disc and a 0.8mm dia hole was drilled near the edge of the disc. The electrode was polished with 600 SiC W/P paper, then 7μm diamond paste followed by 1μm diamond paste. The electrode was then rinsed in D_2O and acetone. The final stage of the electrode preparation was to heat it to 750°C for three hours, then cool for 75 minutes both under a blanket of helium. The electrode was then stored under D_2O until the cell was assembled at which time platinum wire leads were attached through the 0.8mm holes.

The platinum anodes were made from 52 mesh gauze (0.1mm wire dia) supported on a 0.75mm diameter platinum wire to form a cylinder 2.2cm diameter x 3 cm high.

3.2 Cell and Electrolyte

The body of the cell was made from a 125mL high density polyethylene (HDPE) bottle of diameter 5 cm cut to a height of 7 cm. A number 10 black rubber stopper was used as a seal at the top of the cell. Holes were made through the stopper to accommodate the recombiner, helium purge gas inlet, type T thermocouple, sample port and electrodes. The HDPE bottles and stoppers were pretreated in a 1M LiOD solution in a beaker on a hot plate for six hours at 50°C, washed with deionized water and placed in an oven at 110°C for four hours before being placed in a desiccator.

A stock solution of 3 Molar LiOD was prepared by dissolving 25g of lithium metal in D_2O under a blanket of argon. This was diluted to make a significant quantity of 1M and 0.1M electrolytes for the two cells to last the duration of the tests.

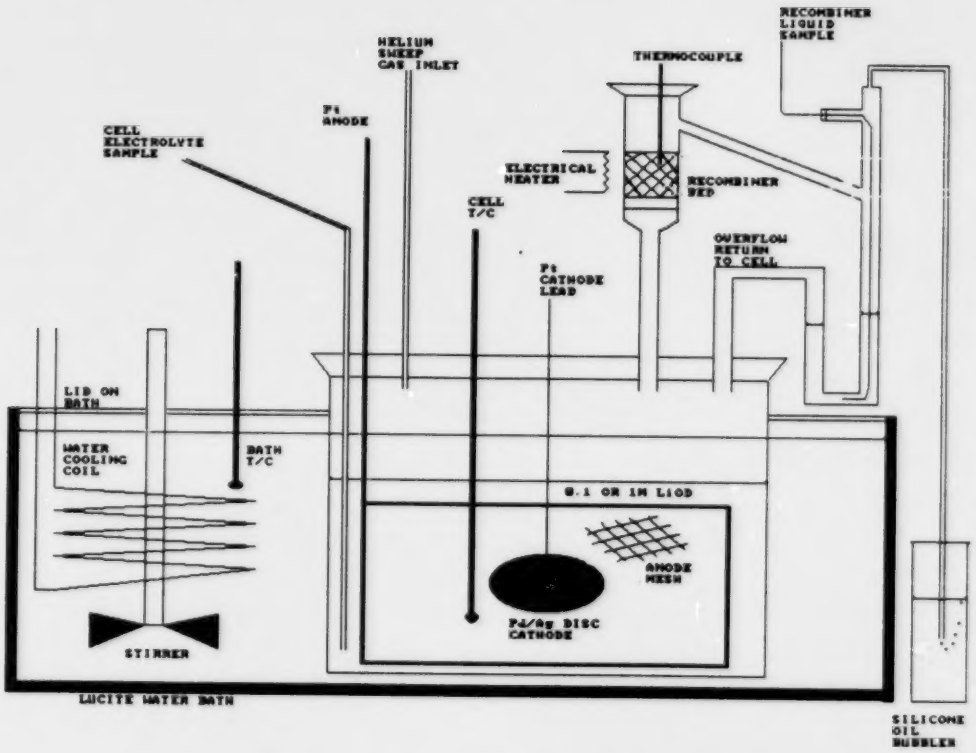

FIGURE 1

SCHEMATIC OF EXPERIMENTAL EQUIPMENT

3.3 Other Equipment

The recombiner bed (about 2.5cm dia x 5 cm long) contained 25% by weight palladium on alumina beads from United Catalysts. The catalyst was heated to remove light water before use. It was necessary to modify the recombiner whereby liquid was returned directly to the cell and not through the recombiner gas riser. This reduced carryover of liquid into the recombiner. Entrainment was dependent on cell level and reduced at lower cell levels.

The cells were placed in a bath kept cool by tap water to maintain a temperature of about 7 to 8°C. Deionized water evaporated from the bath was replaced on a daily basis.

A low flow of helium gas was maintained through each cell and exited through a silicone oil bubbler. This minimized carbon dioxide and light water back diffusion from the surrounding air.

A BK Precision DC power supplies (Model1635) were used to maintain constant current through each cell. A strip chart recorder was used to monitor the voltage as well as the cell temperature.

A data logging system was added for continuous measurement of the cell voltage, the cell current, the cell temperature given by a glass shielded thermocouple, and ambient temperature.

3.4 Radiation Measurements

A neutron counter (Bicron) was set up with a detector to monitor fast neutrons. It used an organic scintillant/photomultiplier combination technique and was installed about 10cm from the cells to monitor both cells simultaneously.

Tritium measurements were carried out on 1 mL samples which were treated with a scintillant recommended for high ionic concentrations to avoid errors due to chemiluminescence.

4.0 RESULTS AND DISCUSSION

Detailed results for cells 1 to 4 are given in Appendix A and B. The basic procedure was to charge the cathode with deuterium at low current density for several days and then run continuously at high current density for several months. About one month before the end of the tests, the electrode surface was poisoned with tetrabutyl ammonium hydroxide and thiourea in an attempt to increase the deuterium content of the electrode. One of the cells was cooled to close to 0°C to investigate the effect of a temperature change.

4.1 NEUTRON COUNTS

The neutron count rate (background averaged over twenty-four hours and expressed as counts/min, see Figure A.1) tended to increase slightly during the test period and fluctuate by ±5%.

As shown in Table 1, the neutron signal exceeded the background by about three standard deviations on six occasions. In all except one instance, these were spontaneous and not as a result of a deliberate action at the particular time to induce an effect. A typical example is given in Figure 2 and the neutron counts on the other days are graphed in Appendix A. Checks with the National Research Council indicated that there was no high solar activity/flares during these six instances.

Nearing the end of the test program, the water bath was cooled with ice to investigate the effect of temperature so it was necessary to stop and start the stirrer to prevent damage. It was found that the neutron detector recorder output was triggered during these stop/start operations. Previous to this discovery, it was not necessary to stop the stirrer or other nearby electrical equipment so we think there is not a strong likelihood of stirrer start/stop operations being responsible for the signals. Also, three of the incidences occurred outside of working hours so it is not likely that stirrer start/stops were responsible. However, it has been reported that neutron detectors are sensitive to line interferences so we cannot rule out electrical line interference as a possible cause of the high signals during the six instances.

There was also further evidence of the capricious nature of cold fusion tests. A significant neutron signal was obtained just after adding poison to a cell on one day but on the following day, another significant signal was obtained just before adding poison. Figures A.2 and A.3 show that the excursions occurred at the same time on two successive days; we have no explanation for this.

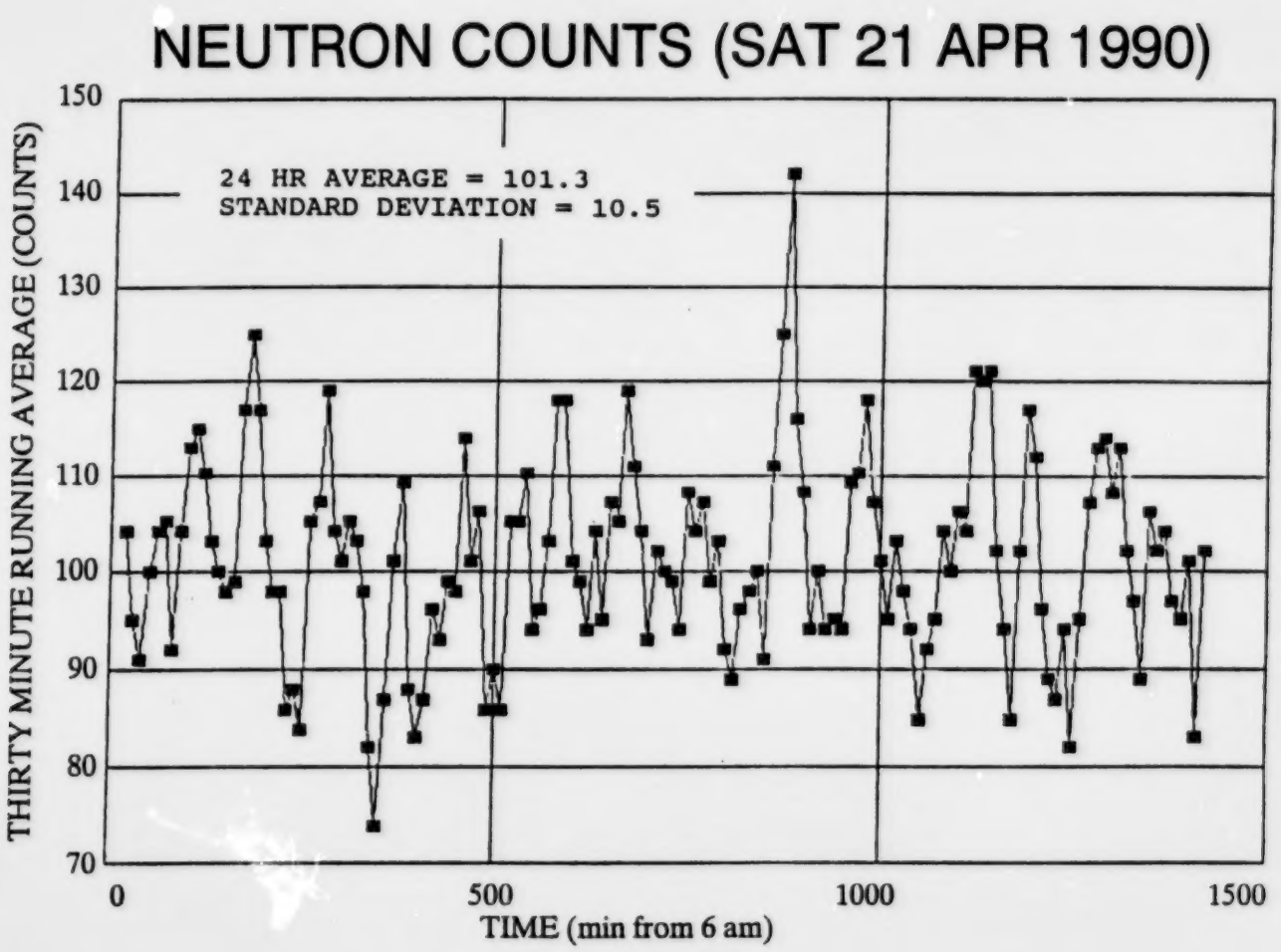

FIGURE 2. HIGH NEUTRON COUNTS OVER A THIRTY MINUTE PERIOD

Table 1. Significant Neutron Counts during Tests.

DATE	TIME (MIN)	COUNTS/ TIME INTERVAL	STANDARD* DEVIATIONS ABOVE BACKGROUND/ (STD DEVIATION)
14FEB 1990	18:00	142/30	3.6/(10.3)
20MAR	11:40	135	2.9/(10.3)
21MAR	10:30	143/30	3.7/(10.3)
6APR	22:00	394/90	4.5/(17.7)
17APR	09:20	60/10	4.2/(5.9)
21APR	20:30	142/30	3.6/(10.3)

*Standard deviation estimated from data of many days.

4.2 TRITIUM ACTIVITY

The tritium concentration in the cells and recombiners were tracked with time as the tests proceeded. As a check, concentration determinations were also frequently carried out on samples of the make-up electrolyte batch which was prepared at the start of the tests. It was found that reproducible results were obtained at the low molarity levels employed in the cells but there were occasional high values of the background tritium content of the make-up electrolyte. As shown in Figure 3, there was no evidence of tritium production for the 1.0 molar LiOD cell nor was significant tritium produced in the 0.1 molar cell. Additional graphs are given in Appendix A.

4.3 OTHER RESULTS/TESTS

No large unexplained temperature excursions occurred. In general, the cell temperature was affected by the temperature of the make-up water to the bath, the bath level, the cell level and whenever the cell was sampled and poisons were added.

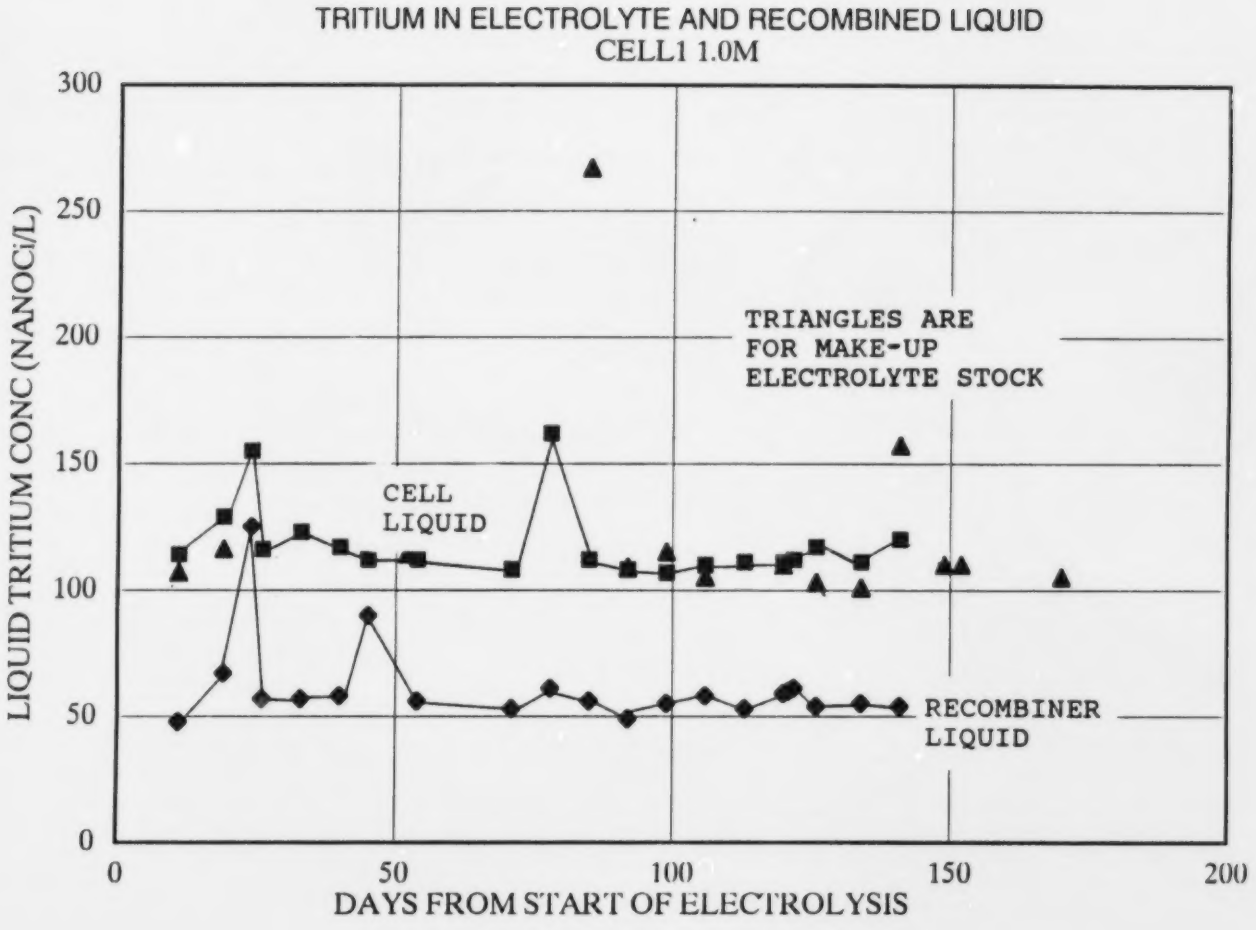

FIGURE 3. THERE WAS NO EVIDENCE OF TRITIUM PRODUCTION AND TRITIUM WAS DEPLETED IN THE CELL OFF GASES/RECOMBINED LIQUID

A 50mL cell consisting of a 2.5cm long palladium wire cathode, a platinum anode and 1.0M LiOD was set up with a flow of helium through the cell to a recombiner. The cell current was cycled on and off for various times and between high and low current densities. No significant tritium was generated during these tests nor after the cell was poisoned with the use of tetrabutyl ammonium hydroxide.

Another test consisted of charging a palladium wire with deuterium and then adding some cupric sulfate to the electrolyte to plate copper onto the Pd cathode. After plating, the wire was removed from the cell and immediately dropped into glycerol at 200°C. Neutron generation was not significant for the rapid heating test nor was it significant when the wire was dropped into liquid nitrogen. Also, heating the wire very quickly to over 1000°C with a laser did not generate neutrons above background.

5.0 CONCLUSIONS

No excess tritium production was found in these tests. There was weak evidence of neutron production but the results are inconclusive. The frequency and low levels of neutron generation, if any, suggest a low probability of usefulness in practice.

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APPENDIX A Detailed Experimental Data

Details of the neutron count rate, the cell current and voltage, and various operations are given in Table 1.

Neutron data are given in Figures A.1 to A.6. Neutrons were counted for ten minute intervals and the data were used to generate averages over longer time intervals in an attempt to reduce the variability of the data. To generate a running average over a given time period, the oldest ten minute value was dropped and the latest added.

The tritium concentration data are given in Figures A.6 and A.7 (see also Figure 3). In general, reproducibility of the measurements was excellent for the 0.1M feed electrolyte but difficulties increased as the molarity was increased.

It is normally found during the electrolysis of different hydrogen isotopes that the cell off-gases are depleted in tritium. Hence, as expected, the recombined liquid contained a lower tritium concentration (about 25%) than the cell electrolyte. In Figure A.7, the cell liquid tritium content increased during the first 50 days of operation because of difficulties with entrainment of electrolyte up the recombiner riser. This resulted in flash evaporation and loss of low tritium-containing gases with the helium flow out of the system and possibly condensation of high tritium-containing liquid in the recombiner. Hence, it was necessary to add electrolyte to the cell to make up for the lost gases resulting in a buildup of tritium in the cell. The entrainment and flash evaporation explains the increase in tritium at day 25 in the recombined liquid with no increase seen in the cell electrolyte. These numbers are small and nowhere near the four orders of magnitude or more concentration factors reported by Bockris(1990) and Iyengar and Srinivasan(1990). After this period, the cell and recombiner liquid tritium concentration remained relatively constant.

TABLE A.1 SUMMARY OF OBSERVATIONS FOR CELLS 1 AND 2
IF NOT GIVEN, PARAMETER VALUE IS LAST PREVIOUS VALUE

DATE	DAY	BATH TEMP DEG C; NEUTRON COUNT RATE	COMMENTS CELL1 1.0M LiOD	COMMENTS CELL2 0.1M LiOD
MON 20NOV 1989	1	10 DEG C 3.4	1.984G Pd/Ag CATHODE; ELECTROLYSIS STARTED; 83mL OF 1.0M LiOD; 200mA CURRENT	CELL 2 OPERATION BEGINS AT DAY 2
21NOV	2	3.46 AVG OVER 24H	200mA; 3.7V; RECOMBINER TEMP 180-200 DEG C	1.9600G Pd/Ag CATHODE; ELECTROLYSIS STARTED; 83mL OF 0.1M LiOD; 200mA, 4.7V
22NOV	3	3.43/24H	200mA; 3.5V	200mA, 4.8V; RECOMBINER 180-190 DEG C
23NOV	4	3.37/24H		
24NOV	5	3.38/24H		
27NOV	8	9.5 DEG C 3.32/72H	TUBE FROM CELL TO RECOMBINER CONTAINED ENTRAINED LIQUID ELECTROLYTE WHICH WAS RETURNED TO CELL	TUBE FROM CELL TO RECOMBINER CONTAINED ENTRAINED LIQUID ELECTROLYTE WHICH WAS RETURNED TO CELL
28NOV	9	3.35/24H	SAME AS ABOVE	
29NOV	10	3.34/24H	RETURN ENTRAINMENT TO CELL	
30NOV	11	3.29/24H	RETURN ENTRAINMENT TO CELL SAMPLE CELL AND RECOMBINER FOR TRITIUM	200mA, 4.9V RETURN ENTRAINMENT TO CELL SAMPLE CELL AND RECOMBINER FOR TRITIUM
1 DEC	12	3.30/24H	200mA; 3.5V	200mA, 4.8V RETURN ENTRAINMENT TO CELL
4 DEC	15	3.30/72H	INCREASE CURRENT TO 2A; 6.2V; RETURN ENTRAINMENT TO CELL	200mA, 4.9V
5 DEC	16	3.29/24H	ADD 3 mL HEAVY WATER TO CELL	INCREASE CURRENT TO 2A, 14.0V
6 DEC	17	9 DEG C 3.5/24H		2A, 14.3V
7 DEC	18	3.3/24H	2A; 6.1V; ADD 5mL D2O TO CELL	ADD 5mL D2O TO CELL
8 DEC	19	3.26/24H	2A; 6.2V; ADD 5mL D2O TO CELL; WHITE DEPOSIT FORMING IN RECOMBINER	2A, 14.6V; ADD 5mL D2O TO CELL
11DEC	22	3.35/72H	2A; 8.0V; ADDED 20mL D2O TO CELL	2A, 16.9V; ADD 12 mL D2O TO CELL
12DEC	23	3.38/24H	1.75A; 29.8V; REMOVED 7.4mL FROM RECOMBINER; ADDED 3mL 3M LiOD TO CELL; CURRENT 2A; 15.3V; RECOMBINER 243 DEG C; REDUCED CURRENT 0.6A; 6.8V	2A, 21.0V; CURRENT REDUCED TO 0.6A, 8.9V RECOMBINER 212 DEG C
13DEC	24	8 DEG C 3.39/24H	CELL OFF; REASSEMBLED WITH MODIFIED RECOMBINER (LIQUID RETURNED DIRECTLY TO CELL; 75mL OF NEW 1.0M LiOD USED + 3mL OF D2O INTO RECOMBINER RETURN TUBE; CELL BACK ON AFTER 3H AT 0.6A; 3.4V AFTER 2H, INCREASE TO 2A; 5.8V	0.6A, 12.6V; RECOMBINER 230 DEG C
14DEC	25	3.30/24H	2A; 6.1V; RECOMBINER 225-250 DEG C	CELL OFF; MODIFIED RECOMBINER INSTALLED; 75mL OF 0.1M LiOD ADDED + 3mL D2O TO RECOMBINER RETURN TUBE; CELL BACK ON AFTER 2.75H AT 0.2A, 4.3V; AFTER 1H INCREASE TO 2A, 18.5V
15DEC	26	3.51/24H	SAMPLED FOR TRITIUM	2A, 14.2V; RECOMBINER 225-250 DEG C SAMPLED FOR TRITIUM; LEAKY RUBBER LID REPLACED; NEW 70mL 0.1M LiOD USED; 2A, 14.4V
18DEC	29	3.33/24H	2A, 6.0V	2A, 18.4V
19DEC	30	3.35/24H		
20DEC	31	3.41/24H		2A, 18.8V
21DEC	32	3.32/24H	2A, 6.1V	
22DEC	33	3.35/24H		
27DEC	38	7 DEG C 3.39/120H	2A, 6.2V	2A, 19.0V; ADDED 5mL 0.1M LiOD; 2A, 18.8V
28DEC	39	3.33/24		2A, 19.3V
29DEC	40	3.31/24H	SAMPLED FOR T	SAMPLED FOR T; ADDED 3mL OF 0.1M LiOD
31DEC	42	3.33/48H	TWO HOUR POWER FAILURE OVERNITE; 2A, 6.1V	2A, 19.8V

DATE	DAY	BATH TEMP DEG C; NEUTRON COUNT RATE	COMMENTS CELL1 1.0M LiOD	COMMENTS CELL2 0.1M LiOD
2JAN 1990	44	7 DEG C 3.40/48H	2A, 10.3V; REMOVED 3mL ELECTROLYTE TO LOWER LEVEL IN CELL TO STOP ENTRAINMENT IN GAS RISER	2A, 21.6V; ADDED 7.5mL OF 0.1M LiOD ENTRAINMENT STILL PRESENT
3JAN	45	3.43/24H	2A, 13.1V; REMOVED 4mL FOR ANALYSIS AND TO LOWER LEVEL IN CELL	SAMPLED FOR T
4JAN	46	3.35/24H	2A, 13.0V	2A, 21.4V; ADD 3mL 0.1M LiOD
5JAN	47	3.33/24H	2A, 13.4V; 5mL REMOVED TO LOWER LEVEL	2A, 20.8V; ADD 3mL 0.1M LiOD TO CELL
8JAN	50			2A, 21.8V; ADD 3mL OF 1.0M LiOD TO INCREASE VOLUME & DECREASE VOLT TO 15.5V
9JAN	51	3.33/24H	REMOVED 20mL FROM CELL, ADDED 10mL OF 3.0M LiOD; VOLTAGE REDUCED TO 7.1V	2A, 15.5V;
10JAN	52	3.37/24H	2A, 7.7V	2A, 15.2V; ADD 3mL OF 0.1M LiOD
11JAN	53	3.37/24H		2A, 15.3V;
12JAN	54	3.41/24H	2A, 7.6V; SAMPLED FOR T	2A, 14.9V; SAMPLED FOR T; ADDED 6mL 0.1M LiOD TO INCREASE LEVEL
15JAN	57	3.38/72H	2A, 15.7V; REMOVED 18mL ELECTROLYTE ADDED 10mL OF 3M LiOD, 2A, 7.9V; He FLOW OFF	2A, 15.2V; ADDED 12mL OF 0.1M LiOD TO INCREASE LEVEL; He FLOW OFF
16JAN	58	3.31/24H	2A, 7.9V	2A, 15.3V
17JAN	59	3.29/24H		
18JAN	60	3.48/24H	2A, 8.0V; He FLOW ON	He FLOW ON
19JAN	61	3.37/24H	SAMPLE FOR T	2A, 15.8V
22JAN	64	3.36/72H		2A, 16.9V
23JAN	65	3.40/24H		2A, 17.2V
24JAN	66	3.39/24H		2A, 17.3V
25JAN	67	3.41/24H	2A, 7.8V	
26JAN	68	3.56/24H	2A, 7.7V	
29JAN	71	3.41/72H	SAMPLED FOR T	SAMPLED FOR T; ENTRAINMENT HAS STOPPED
30JAN	72	3.34/24H		SAMPLED RECOMBINER FOR T
31JAN	73	3.38/24H		
1FEB	74	3.38/24H		2A, 17.7V
2FEB	75	3.36/24H		2A, 17.5V
5FEB	78	3.34/72H	SAMPLED FOR T	2A, 17.6V; SAMPLED FOR T
6FEB	79	3.34/24H		2A, 17.4V
7FEB	80	3.50/24H		2A, 17.5V
9FEB	82			2A, 17.7V
12FEB	85	3.47/72H	SAMPLED FOR T	2A, 17.9V; SAMPLED FOR T
13FEB	86	3.50/24H		
14FEB	87	3.54/24H		
15FEB	88	3.41/24H		
16FEB	89	3.47/24H		2A, 18.2V
19FEB	92	3.42/72H	2A, 7.7V; SAMPLED FOR T; ADDED 1mL 3M LiOD; 2A, 7.3V	2A, 18.1V; SAMPLED FOR T ADD 1mL OF 1M LiOD, 16.9V
20FEB	93	3.37/24H	2A, 7.3V	2A, 16.7V
21FEB	94	3.42/24H	2A, 7.4V	2A, 16.4V
22FEB	95	3.38/24H		2A, 15.9V
23FEB	96	3.51/24H		2A, 15.4V; LIQUID RISING FROM CELL TO RECOMBINER RETURNED TO CELL; LEAK BETWEEN CELL AND RECOMBINER FIXED; ADD 10mL 0.1M LiOD TO BRING LEVEL UP
26FEB	99	3.45/72H		2A, 15.0V; SAMPLED FOR T
27FEB	100	7 DEG C 17 DEG C 3.41/24H	2A, 7.5V	2A, 15.5V; COOLING WATER TO BATH OFF FOR 24H
28FEB	101	6 DEG C 3.40/24H	2A, 7.4V	2A, 15.8V; LIQUID RISING FROM CELL TO RECOMBINER LOWERED

DATE	DAY	BATH TEMP DEG C; NEUTRON COUNT RATE	COMMENTS CELL1 1.0M LiOD	COMMENTS CELL2 0.1M LiOD
1MAR 1990	102	7 DEG C 3.38/24H	2A, 7.4V;	2A, 16.3V; LIQUID RISING FROM CELL TO RECOMBINER LOWERED
2MAR	103	3.40/72H		2A, 17.1V; LIQUID RISING FROM CELL TO RECOMBINER LOWERED
5MAR	106	3.48/24H	2A, 7.3V; SAMPLED FOR T	2A, 18.2V; SAMPLED FOR T; LIQUID RISING FROM CELL TO RECOMBINER LOWERED
6MAR	107	3.42/24H		2A, 18.7V; LIQUID RISING FROM CELL TO RECOMBINER LOWERED
7MAR	108	3.44/24H		
8MAR	109	3.38/24H		
9MAR	110	3.35/24H		
12MAR	113	8 DEG C 3.45/24H		2A, 18.9V; LIQUID RISING FROM CELL TO RECOMBINER LOWERED; 2mL REMOVED FROM CELL; ADDED 2 mL 3M LiOD
13MAR	114	3.43/24H	2A, 7.2V	2A, 12.8V; LIQUID RISING FROM CELL TO RECOMBINER LOWERED
14MAR	115	3.52/24H		2A, 13.6V; LIQUID RISING FROM CELL TO RECOMBINER LOWERED
15MAR	116	3.42/24H		2A, 14.3V; LIQUID RISING FROM CELL TO RECOMBINER LOWERED
16MAR	117	3.45/24H		
19MAR	120	3.46/24H	2A, 7.1A	2A, 14.1V
20MAR	121	9 DEG C 3.50/24H	2A, 7.1V; ADD 0.5mL TETRABUTYL AMMONIUM HYDROXIDE(TBAH)	
21MAR	122	3.46/24H	2A, 8.2V; ADD 0.5mL TBAH	
22MAR	123	3.40/24H	2A, 10.1V	
23MAR	124	10 DEG C 3.46/24H	2A, 10.1V	2A, 14.2V
26MAR	127	3.40/72H	2A, 11.0V	
27MAR	128	3.42/24H		
28MAR	129	3.35/24H		
29MAR	130	3.36/24H	2A, 11.1V	
30MAR	131	3.34/24H		2A, 14.3V
2APR	134	3.46/24H	2A, 11.3V	
3APR	135	3.63/24H	2A, 11.1V; WHITE DEPOSIT ON RECOMBINER RISER TUBE AND RECOMBINER BEADS	
4APR	136	3.56/24H	2A, 11.2V	
5APR	137	3.60/24H	2A, 11.3V;	
6APR	138	11 DEG C 3.42/24H		2A, 14.5V
9APR	141	3.46/24H	SAMPLED FOR T; LIQUID RISING IN RECOMBINER TUBE	SAMPLED FOR T
10APR	142	12 DEG C 3.41/24H		
11APR	143	3.48/24H	2A, 11.1V; SAMPLED FOR T; ADDED 10mg THIOUREA	2A, 14.4V
12APR	144	3.47/24H	2A, 10.9V	
17APR	149	13 DEG C 3.44/24H	2A, 10.6V; SAMPLED FOR T	SAMPLED FOR T
18APR	150	3.44/24H	2A, 10.4V; SAMPLED FOR TRITIUM REMOVED CELL 1 FROM BATH; 2A, 7.8V	
19APR	151	3.39/24H	2A, 6.9V; SAMPLED FOR T; CELL OFF!!!!! PLACED CATHODE BETWEEN FILM FOR FIVE DAYS=> NO FILM FOGGING	ADD 0.01ml TBAH; VOLTAGE INCREASED TO 17.9; DROPPED TO 16.7 AFTER 2 H
20APR	152	3.46/24H		2A, 17.1V; SAMPLED FOR T; 2A, 17.6V
23APR	155	15 DEG C 3.45/24H	ANALYSIS OF CELL1 DEPOSIT BY XRF/XRD/ICP XRD CRYSTALLINE PHASE PRESENT= LITHIUM CARBONATE	2A, 18.1V; SAMPLED FOR T
24APR	156	3.57/24H	NOT FOUND= LiPtH(SUB 0.66) NOT FOUND= LiPtD(SUB 0.66)	
25APR	157	3.49/24H	NOT FOUND= Na(SUB 2)PtD(SUB 4) CARBONATE AS CO3= 76% BY WT	
26APR	158	16 DEG C 3.55/24H	Li=18000PPM, Pt=4600PPM, Si=4000PPM Ca=1100PPM, Pd=427PPM, Al=367PPM Ni=213PPM, Fe=111PPM, Zn=93PPM Cu=32PPMV, Na=18PPM	
27APR	159	3.38/24H		
30APR	162	3.45/24H		2A, 18.1V; SAMPLED FOR T

DATE	DAY	BATH TEMP DEG C; NEUTRON COUNT RATE	COMMENTS CELL1 1.0M LiOD	COMMENTS CELL2 0.1M LiOD
1MAY	163	16 DEG C 3.58/24H	OFF ANALYSIS OF CELL 1 ELECTROLYTE: TOTAL INORGANIC CARBON = 1300 PPM	2A, 18.1V; REDUCED CURRENT TO 0.05A, 3.6V; ADD 10mg THIOUREA
2MAY	164	17 DEG C 3.35/24H	LIGHT WATER < 2% ICP=> Na 22PPM, Al 9PPM, Sn 3PPM, Ca 0.6PPM, Pt 0.3PPM, Pd <.1PPM	0.05A, 3.8V; ADD 10 mg THIOUREA; INCREASE CURRENT TO 2A, 24.2V DOWN TO 21V IN 3 MIN; 2A, 19.2V STEADY
3MAY	165	17 TO 2 DEG C 3.33/24H		2A, 19.1V; SAMPLE FOR T; REMOVE 5mL FROM CELL 2 AND ADD 10mL OF 3M LiOD; 2A, 7V; COOLING WATER OFF; 2A, 7.6V; STOP STIRRER, STIR WITH AIR BUBBLING; COOL BATH TO 2 DEG C WITH ICE; SAMPLE FOR TRITIUM
4MAY	166	2 DEG C		2A, 7.5V; NEUTRON DETECTOR TRIGGERED BY STIRRER ON/OFF SWITCH
7MAY	169	32(!!) DEG C 3.49/72		2A, 6.3V; SAMPLED FOR T; REDUCE CURRENT TO 0.1A, 2.6V COOLING WATER ON; STIRRIER ON
8MAY	170	13 DEG C 3.61/24H		0.1A, 3.5V; SAMPLED FOR T; CELL OFF!!!!!! TAPED ELECTRODE TO FACE OF NEUTRON DETECTOR
9MAY	171	3.48/24H		NO SIGNIFICANT NEUTRON EXCESS SEEN; PLACED ELECTRODE BETWEEN POLAROID INSTANT SHEET FILM FOR 20MIN => NO FOGGING OCCURRED
11MAY	173	3.54/24H		SIMILARLY, AFTER ONE MONTH OF EXPOSURE, NO FOGGING SEEN
14MAY	176	3.47/24H		
15MAY	177	3.51/24H		
16MAY	178	3.51/24H		
17MAY	179	3.49/24H		
18MAY	180	3.61/24H		
22MAY	184	3.51/24H		
23MAY	185	3.49/24H		
24MAY	186	3.49/24H		
25MAY	187	3.53/24H		
28MAY	190	3.52/24H		
29MAY	191	3.44/24H		
30MAY	192	3.60/24H		
31MAY	193	3.53/24H		
1JUN	194	3.65/24H		
4JUN	197	3.54/24H		
5JUN	198	3.57/24H		
6JUN	199	3.38/24H		
7JUN	200	3.51/24H		
8JUN	201	3.49/24H		
11JUN	204	3.46/72H		

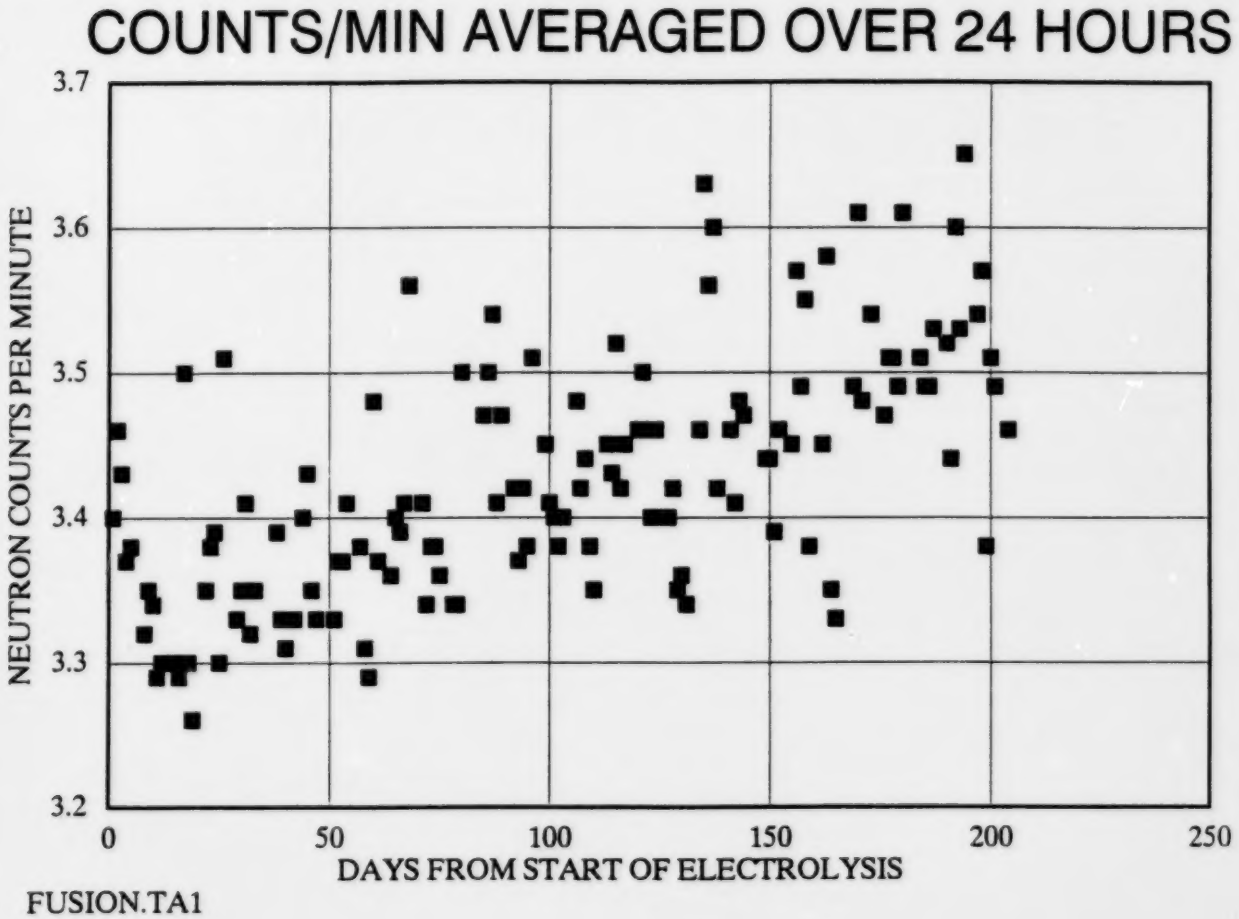

FIGURE A.1 THE AVERAGE COUNT RATE INCREASED BY ABOUT 10% OVER THE 200 DAY TEST PERIOD

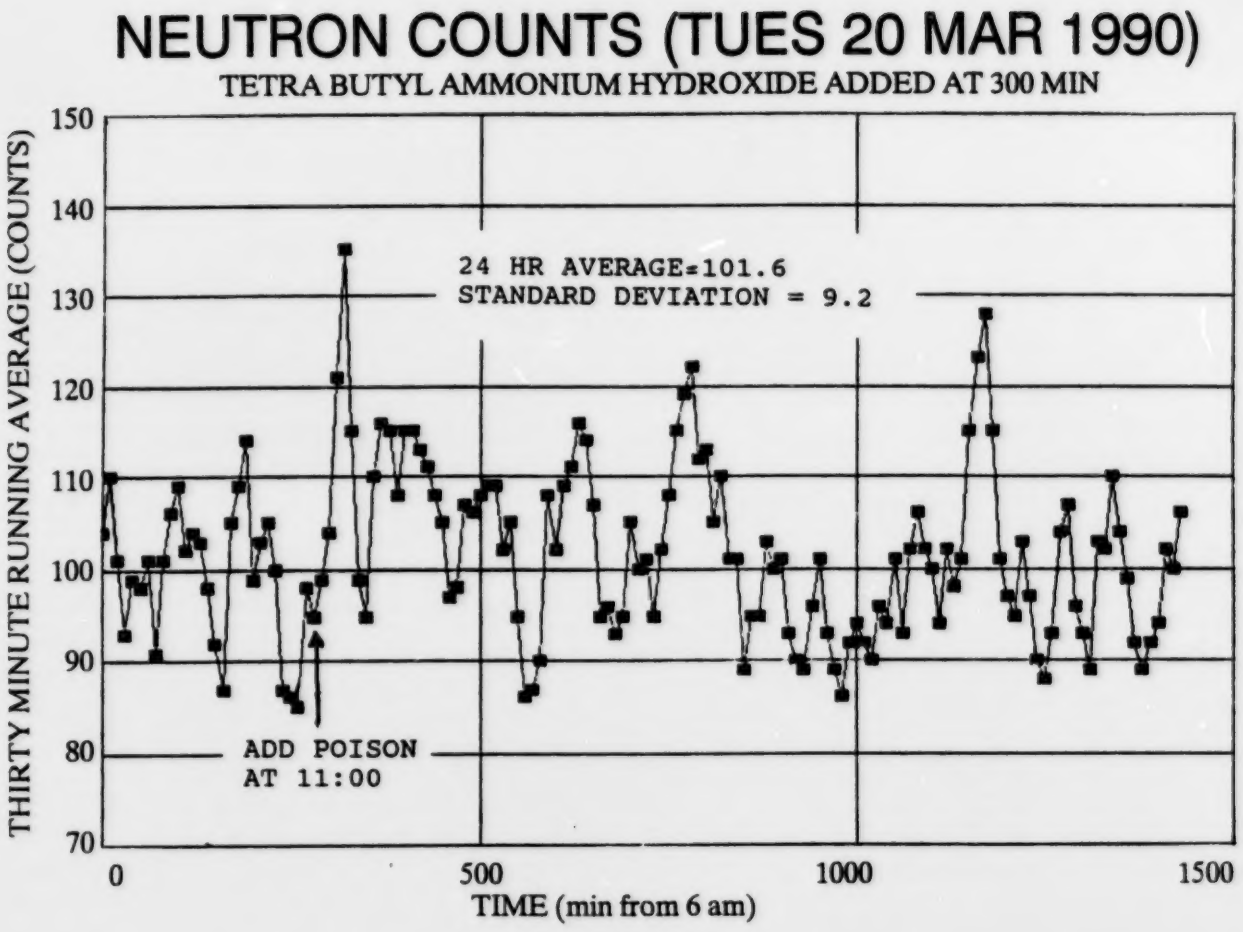

FIGURE A.2 APPARENT INCREASE IN NEUTRON COUNTS WITH POISONING

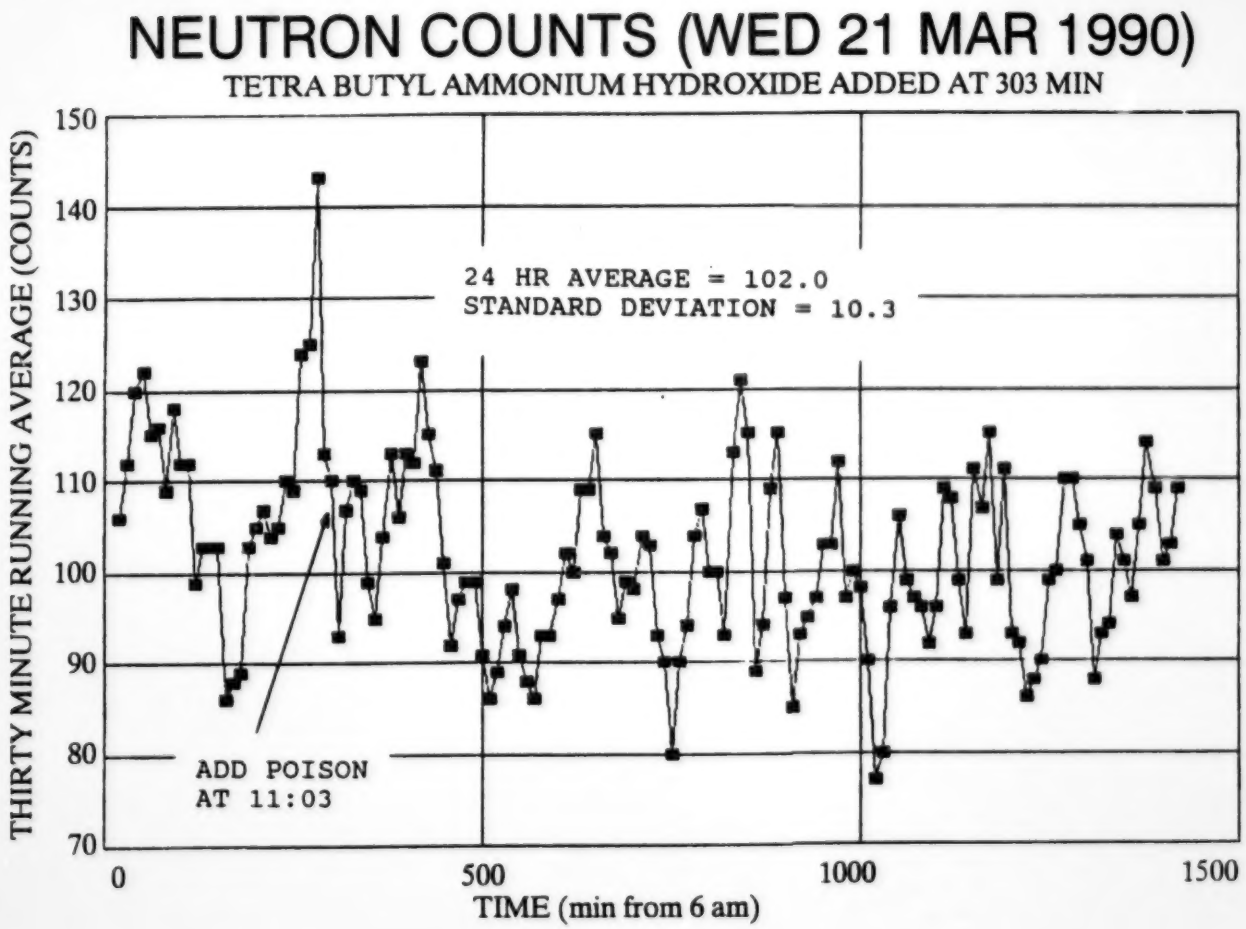

FIGURE A.3 INCREASE IN NEUTRON COUNTS BEFORE POISONING

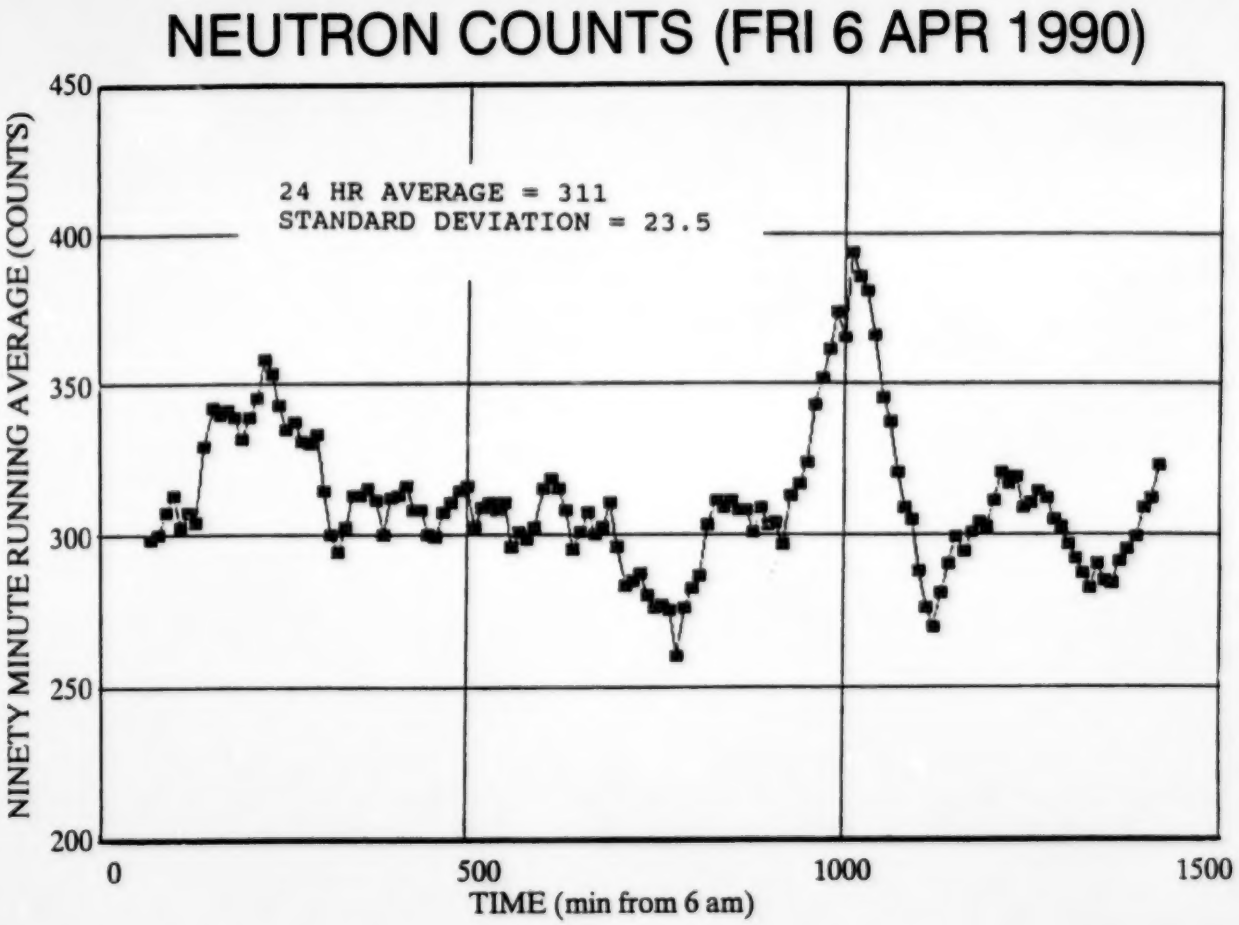

FIGURE A.4 EXTENDED PERIOD OF HIGH NEUTRON COUNTS

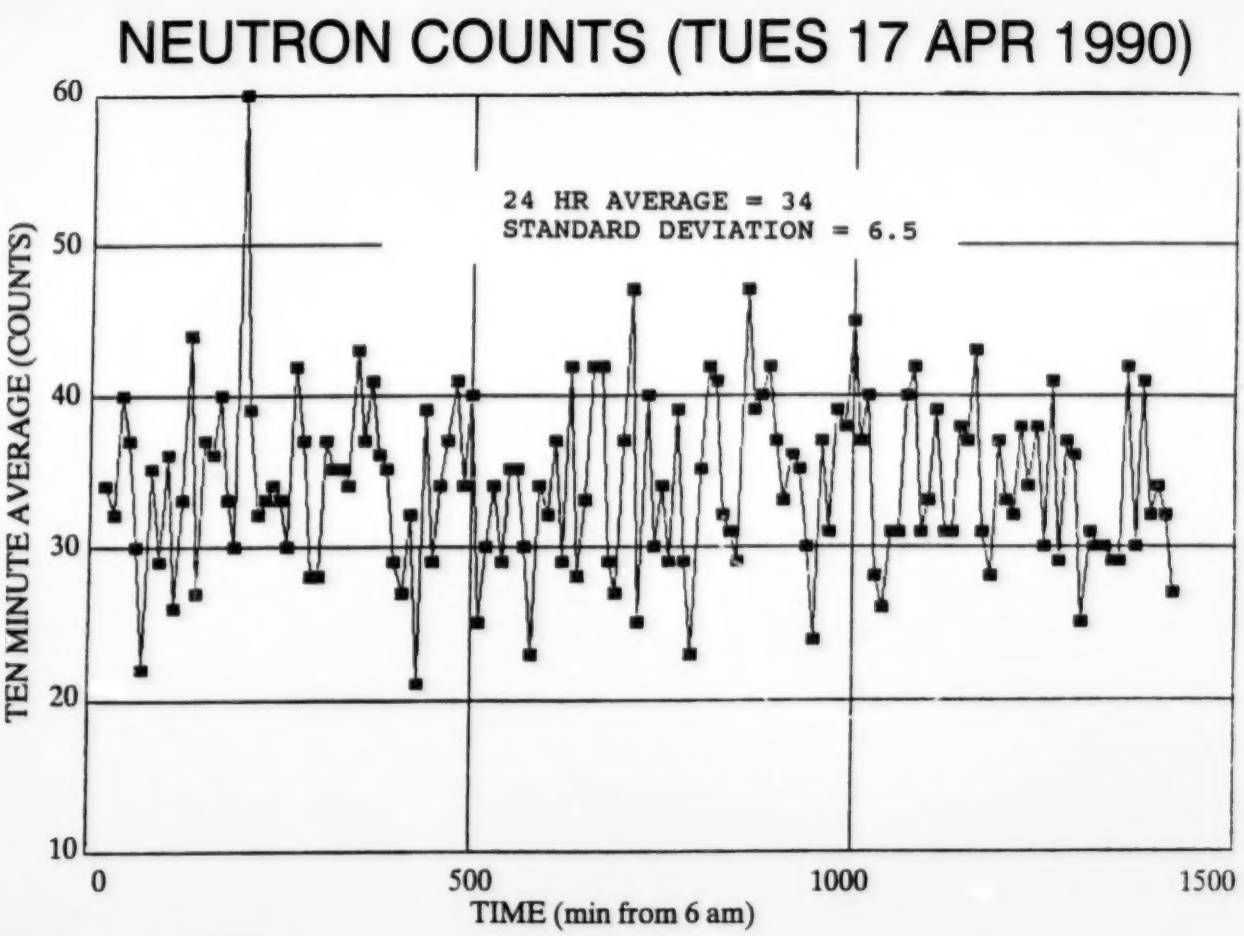

FIGURE A.5 HIGH NEUTRON COUNTS OVER A TEN MINUTE PERIOD

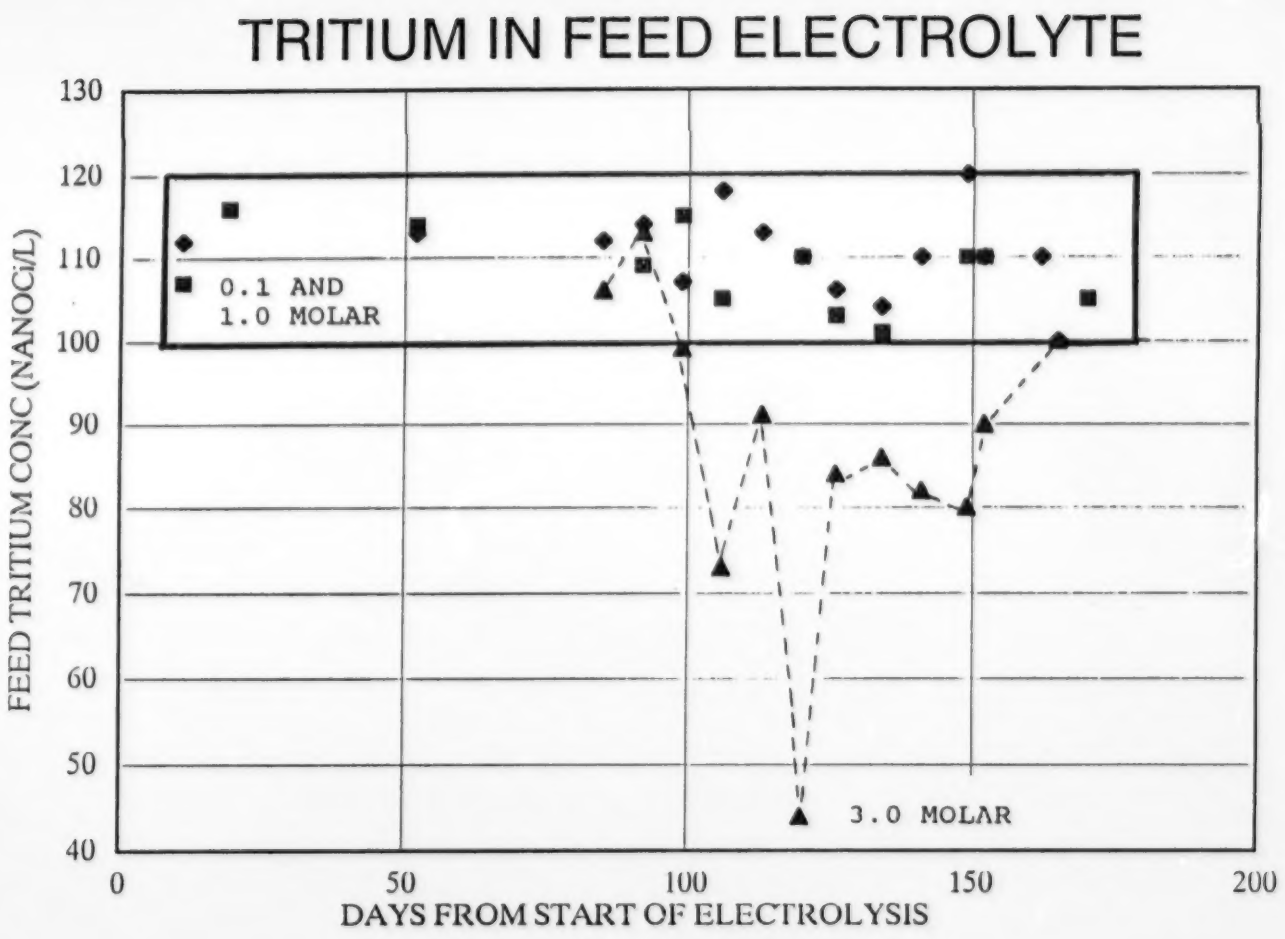

FIGURE A.6 TRITIUM ANALYSES OF 0.1 AND 1.0 M LiOD ELECTROLYTE ARE REPEATABLE BUT VARIABILITY AND LOWER VALUES ARE OBTAINED WITH 3.0M LiOD

NOTE: FOR THE 1.0M LiOD, VALUES OF 267 AT DAY 85 AND 155 AT DAY 141 ARE BELIEVED TO BE IN ERROR (OUTSIDE THE THREE STANDARD DEVIATION LIMIT) AND ARE NOT INCLUDED IN THE ABOVE GRAPH

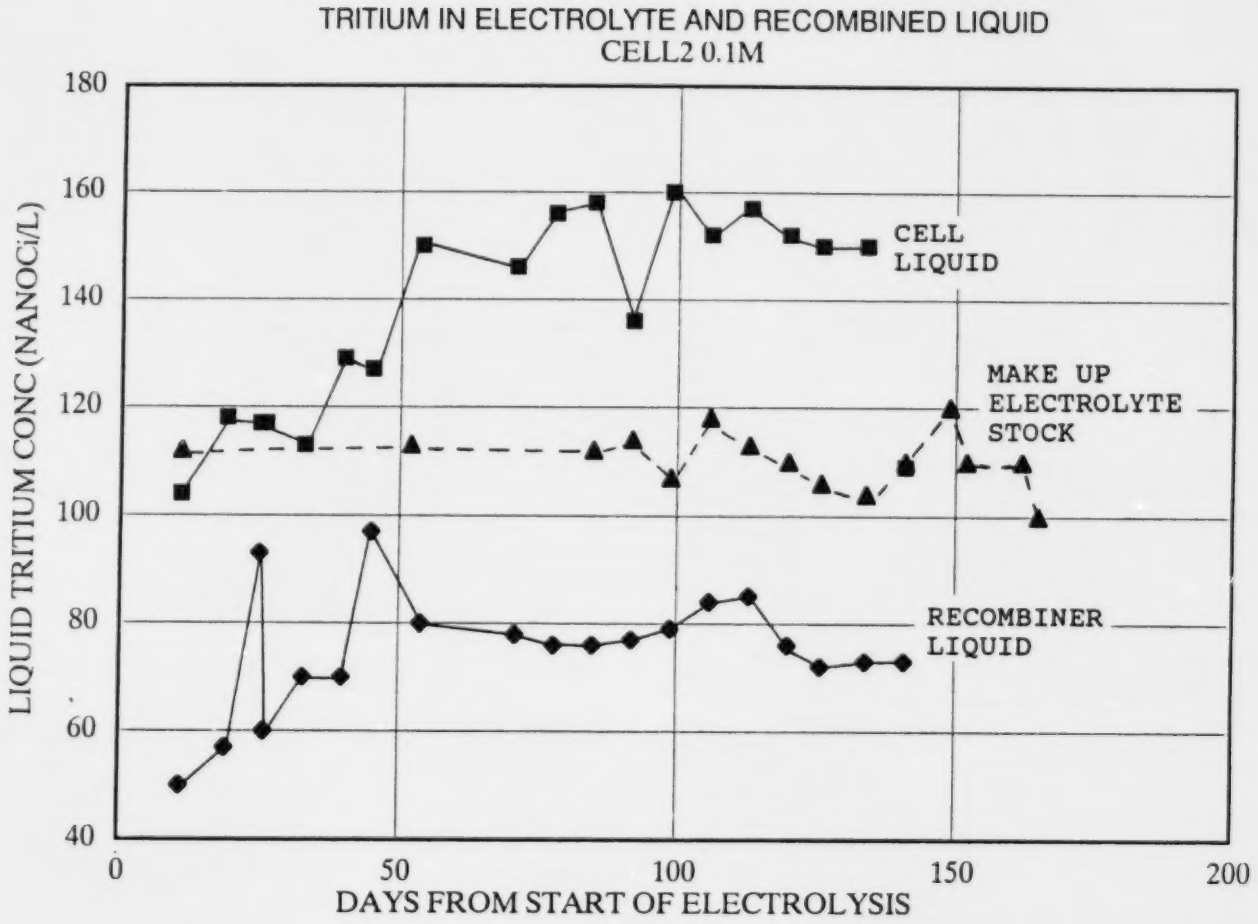

FIGURE A.7 ENTRAINMENT OF CELL LIQUID TO RECOMBINER RESULTED IN ADDITION OF FRESH ELECTROLYTE AND CONCENTRATION OF TRITIUM IN THE CELL ELECTROLYTE AND RECOMBINER LIQUID DURING THE FIRST FIFTY DAYS OF OPERATION